“Study of Molecular Flexibility Operated Mesomorphism”

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Keywords: Thermotropic, Nematic , Smectic , Thermal Stability, Mesomorphism.

Abstract. Homologous series RO-C₆H₄-CH=CH-COO-C₆H₄-CO-CH=CH-C₆H₄-OC₁₄H₂₉(P) of chalconyl novel derivatives have been synthesized and studied with a views to understand and establish the relation between molecular structure and the liquid crystal (LC) properties of thermotropic mesomorphs Series consists of thirteen homologues. Five homologues (C₁ to C₅) are nonliquid crystal (NLC) and the rest of the homologues (C₆ to C₁₈) are enantiotropically nematogenic without exhibition of smectic property. Transition temperatures and textures were determined by an optical polarizing microscope equipped with a heating stage (POM). Transition curves of a phase diagram(N-I and Cr-I/N) behaved in normal manner. N-I transition curve exhibited odd-even effect with negligible abnormality in its behaviour at the C₁₄ homologue. Thermal stability for nematic is 99.75⁰C. The degree of mesomorphism vary minimum 19⁰C at the C₆ homologue to maximum 28⁰C at the C₈ homologue. Analytical and spectral data confirms the molecular structures of the novel homologues. Evaluated thermal data of present novel series are compared with other structurally similar homologous series. Textures of Nematic phase are threded or schlieren.

1. INTRODUCTION

Chalconyl derivatives are well-known for their biological activity as antibacterial, antifungal , antimalarial , anticancer [1,2,3,4,5,6,7] etc. and its involvement in many other biological processes in solution or lyotropic state. Liquid Crystalline (LC) state in thermotropic type is [8] exploited in LC devices [9,10,11]. Present investigation is planned with a view to understand and establish the relation between thermotropic mesomorphism and the molecular structure of a substance. Secondly, the object of the study is also aimed to provide novel LC material of chalconyl derivatives for study to scientiests and technologists working with multidisplinary LC state in the benefit of mankind or living entities including to the biologists and pharmacists. Novel chalconyl derivatives will consists of three phenyl rings bonded through -CH=CH-COO- and -CO-CH=CH- central bridges contributing to total molecular rigidity and two terminal flexible end groups -OR and -OC₁₄H₂₉ which contributes to total molecular flexibility through homologous series in which n-alkyl chain R varies progressively and sequentially from C₁ to C₁₈. The novel compounds will be characterized by analytical and spectral data. Thermometric data will be evaluated and interpreted in terms of molecular rigidity and flexibility [12,13,14,15] depending on molecular structure [16,17,18,19] and then mesomorphic properties and behaviours will be compared with structurally similar series. Number of ester homologous series with or without chalconyl group were studied and reported till the date[20,21,22,23,24,25,26,27,28]. Group efficiency order and structure relation with mesomorphism will be derived.

2. EXPERIMENTAL

Synthesis:

4-n-Alkoxy Cinnamic acids were synthesized by the method followed by Patel and Doshi [29] from 4 - Hydroxy cinnamic acid as prepared by refluxing 4-hydroxy Benzaldehyde and Malonic acid in pyridine and few drops of piperidine. 4- Hydroxy Cinnamic acid was alkylated

using suitable alkylating Agent (R-X) to convert it into 4-n- alkoxy Cinnamic acids (A) prepared by modified method of Dave and Vora [30]. α - 4- hydroxyl benzoyl β -4'- tetradecyloxy phenyl ethylene (B) was prepared by usual established method. Esters were synthesized by a literature method[31]. Thus, the Chalconyl - ester homologue derivatives were filtered and washed with sodium bicarbonate solution and dried and purified till constant transition temperatures using an optical polarising microscope equipped with a heating stage. 4-hydroxy cinnamic acid, Alkyl halides, 4- hydroxy benzaldehyde, Malonic acid, piperidine, 4-hydroxy acetophenone, dicyclohexyl carbodimide, Dimethyl amino pyridine, DCM, MeOH, Acetone required for synthesis were used as received except solvents which was dried and distilled prior to use. The synthetic route to a series is mentioned in Scheme-1.

Scheme 1: Synthetic route to the series

Characterization

Selected members of the novel homologous series were characterized by Elemental Analysis, infra red spectroscopy, ^1H NMR spectra. IR spectra were recorded by Perkin-Elmer spectrum GX, ^1H NMR spectra were recorded on Bruker using CDCl_3 as solvent. Microanalysis was performed on a Perkin-Elmer PE2400 CHN analyzer. Transition temperature and LC properties (Textures) were determined using an optical polarizing microscopy equipped with a heating stage. Textures of nematic phase determined by miscibility method.

Analytical Data:

Table 1: Elemental Analysis for octyloxy, decyloxy, tetradecyloxy and octadecyloxy derivatives

Sr. No.	Molecular formula	%Elements found		%Elements Theoretical	
		C	H	C	H
1	$\text{C}_{46}\text{H}_{62}\text{O}_5$	79.21	8.93	79.31	8.90
2	$\text{C}_{48}\text{H}_{66}\text{O}_5$	79.60	9.08	79.77	9.14
3	$\text{C}_{52}\text{H}_{74}\text{O}_5$	80.35	9.56	80.20	9.51
4	$\text{C}_{56}\text{H}_{82}\text{O}_5$	80.45	9.76	80.57	9.83

IR Spectra in cm^{-1} for Octyloxy & Hexadecyloxy Derivatives:

Octyloxy : 663(Cis -CH=CH-), 713 Polymethylene (-CH₂-)n of -OC₈H₁₇, 831(-C-H- def. m di-substituted-Para), 771 Polymethylene (-CH₂-) of -OC₁₄H₂₉, 937 (-C-H- def. hydrocarbon), 1062 and 1018 (-C-O-) Str, 1168,1253 and 1390 (-C-O str in -(CH₂)n chain), 1446 and 1467 (-C-H- def. in CH₂), 1510 (-C=C-)str, 1602 and 1666 (-C=O group) and (-COO- ester group), 2848 and 2916 (-C-H str in CH₃).

Hexadecyloxy: 663(Cis -CH=CH-), 711 Polymethylene (-CH₂)n of -OC₁₆H₃₃, 831 (-C-H- def. m di-substituted-Para), 769 Polymethylene (-CH₂-) of -OC₁₆H₃₃, 937 (-C-H- def. hydrocarbon), 1062 (-C-O-) Str, 1390, 1165 and 1253 (-C-O str in -(CH₂)n chain, 1452 (-C-H- def. in CH₂),1502 (-C=C-)str, 1600 and 1672 (-C=O group) and (-COO- ester group), 2848 and 2918 (-C-H str in CH₃).

^1H NMR spectra in CDCl_3 in δ ppm for Hexyloxy & Dodecyloxy Derivative:

Hexyloxy : 0.87(t, -CH₃ of -C₆H₁₃), 1.2-1.4(m, n-poly methylene groups of -OC₆H₁₃),1.78 (m, n-poly methylene groups of -OC₁₄H₂₉),3.5-3.7(s,-OCH₂-CH₂-of-OC₆H₁₃),4.01(s,-OCH₂-CH₂-of-OC₆H₁₃) 6.51 (d,-CH=CH-) 6.9-7.4(s,-CO-CH=CH), 8.09 (s, p-disubstituted phenyl ring).

Dodecyloxy : 0.87 (t,-CH₃ of -C₁₂H₂₅), 1.2-1.5(m, n-poly methylene groups of -OC₁₂H₂₅),1.80 (m, n-poly methylene groups of -OC₁₄H₂₉),3.5-3.6(s,-OCH₂-CH₂-of-OC₁₄H₂₉),4.0(s,-OCH₂-CH₂-of-OC₁₂H₂₅),6.47 (d,-CH=CH-) 6.9-7.5(s,-CO-CH=CH),8.1 (s, p-disubstituted phenyl ring).

Table 2: Texture of Nematic Phase of C₇, C₁₀, C₁₄, C₁₆ by miscibility method.

Sr. No.	Homologue	Texture
1	C ₇	Threaded
2	C ₁₀	Threaded
3	C ₁₄	Schlieren
4	C ₁₆	Schlieren

Table 3: Transition Temperature of Homologous series

Sr.no	R= n-alkyl group	Transition temperatures in $^{\circ}\text{C}$		
		Smectic	Nematic	Isotropic
1	C1	-	-	122.0
2	C2	-	-	129.0
3	C3	-	-	134.0
4	C4	-	-	118.0
5	C5	-	-	110.0
6	C6	-	84.0	103.0
7	C7	-	65.0	91.0
8	C8	-	69.0	97.0
9	C10	-	64.0	88.0
10	C12	-	91.0	112.0
11	C14	-	80.0	102.0
12	C16	-	84.0	107.0
13	C18	-	74.0	98.0

Homologues series

Figure 1: Phase behaviours of Series

3. RESULT AND DISCUSSION

α - 4 - Hydroxy benzoyl β - 4' - tetradecyloxy ethylene (m.p. 80 °C) is a nonmesomorphic component, which on condensation with cis n-alkoxy cinnamic acids yielded novel chalconyl derivatives. C₁ to C₅ homologues are nonmesomorphic (NLC), Whereas, rest of the derivatives are (C₆ to C₁₈) enantiotropically nematogenic without exhibition of smectogenic character. Transition temperatures were plotted against the number of carbon atoms present in n-alkyl chain of left n-alkoxy group. Transition curves Cr-I/N and N-I were obtained on linking, like or related points, which showed phase behaviours of series. Cr-I/N transition curve adopted zigzag path of rising and falling with overall descending tendency. N-I transition curve initially rises and then falls with overall descending tendency except negligible abnormality at the C₁₄ homologue. Odd-even effect is observed for N-I transition curve. N-I transition curve for even numbered methylene unit occupied higher position than odd number's. N-I transition curves showing odd-even effect merges into each other at C₉ homologue and then prolonged as a single transition curve for longer and higher homologues from and beyond C₉ homologue. Thus Cr-I/N and N-I transition curves behaved in almost normal manner. Analytical and spectral data confirmed the molecular structures of respective homologues. Thermal stability for nematic is 99.75 and mesomorphic phase length ranges from 19°C to 28°C at the C₆ to C₈ homologue respectively. Thus, thermometric properties

vary from homologue to homologue in present series with changing flexibility due to number of methylene unit or units of left n-alkoxy terminally situated end group, keeping rest of the molecular part unchanged throughout the same series. Thus, series of present investigation is partly nematogenic and of low melting type and relatively short ranged liquid crystallinity as well as low thermal stability.

The exhibition or inexistence of mesomorphic property by a substance depends upon its suitable or unsuitable magnitudes of anisotropic forces of intermolecular end to end and/or lateral forces as a consequence of favourable or unfavourable molecular rigidity and flexibility. The molecular rigidity remains unaltered throughout the series from homologue to homologue in the same series; but, its molecular flexibility alters from homologue to homologue in the same series due to changing number of methylene unit or units present in n-alkyl chain bonded to first phenyl ring through oxygen atom. The combined effect of molecular rigidity and flexibility of suitable magnitude induces mesomorphism in a substance to disalign the molecules and resist exposed thermal vibrations on molecules under microscopic examination under floating condition on the surface. The inexistence or failure of exhibition by C_1 to C_5 homologues is attributed their inability to resist exposed thermal vibrations due to unsuitable magnitudes of intermolecular end to end or/and lateral attractions which abruptly breaks crystal lattices and smoothly transform crystalline state to isotropic state without passing through LC state, due to low dispersion forces and low magnitudes of dipole-dipole and electronic interactions between instantaneous dipole produced by spontaneous oscillations of electron clouds of the molecules leading to high crystallising tendency. Thus, molecules of nonmesomorphs randomly oriented in all possible directions with high order of disorder or uncontrolled movement. Such molecules do not acquire monotropic LC state on cooling the isotropic mass. Thus, any sort of mesomorphism either smectic or nematic is absent for C_1 to C_5 homologues. The exhibition of enantiotropic nematic mesophase formation commencing from C_6 homologue to C_{18} homologue is attributed to disalignment of molecules at an angle ninety or less than ninety degree which resisted exposed thermal vibrations by suitable magnitudes of end to end attractions, acquiring statistically parallel orientational order of molecular arrangement under floating condition during microscopic examination for definite range of temperature according to molecular permanent dipole moment, aromaticity, molecular rigidity, flexibility, polarity and polarizability etc.

However, all the mesogenic or nematogenic (C_6 to C_{18}) homologues fail to exhibit smectogenic character due to absence of lamellar packing of molecules in their preoccupied crystal lattices, which eliminated the possibility of acquiring sliding layered molecular arrangement in floating condition to show smectogenic texture under microscopic (POM) observation, either monotropically or enantiotropically for a single novel homologue. The odd-even effect is observed due to sequentially added methylene unit from C_6 or C_7 homologue. The disappearance of odd-even effect due to merging of N-I transition curves from and beyond C_9 homologue for higher homologues (C_9 to C_{18}) of longer n-alkyl chain is attributed to the coiling, bending or flexing or coupling of n-alkyl chain of left and/or right end groups with the principal axis of core structure. The negligible abnormality appearing at the C_{14} homologue is attributed due to zero difference of group polarities of $-OC_{14}H_{29}$ identical left and right end groups, because the magnitude of vector sums of polarities of all the bonds of $-OC_{14}H_{29}$ are exactly equal and opposite in directions which results into actual effectiveness in the magnitudes of molecular flexibility, reflecting to mesogenic behaviour of C_{14} , quite differently than the rest of the homologues of a present novel series. Changing trends in mesomorphic properties like thermal stability, commencement of mesophase, mesophase length etc. depended upon molecular length which sequentially undergo varied, which undergo changing permanent dipole moment across the long molecular axis, dispersion forces, thermodynamic quantity enthalpy (ΔH), extent of noncoplanarity which related to changing molecular structure which causes the change in the magnitudes of combined effect of molecular rigidity and flexibility. The molecular rigidity due to phenyl rings and central bridges linking them remain unaltered throughout the same series from homologue to homologue, but the molecular flexibility due to difference in polarity of two end groups of present series undergo varied.

Therefore, the magnitudes of their combined effect (rigidity + flexibility) which changes from homologue to homologue in the same series. Thus, thermometric resistivity and suitable magnitudes of intermolecular cohesion energy and consequently mesomorphic tendency of a molecule show variation. The variations in mesomorphic properties of a present novel series are compared with the structurally similar other known homologous series as shown below in figure-2.

Figure 2: Structurally similar series

Homologous series 1 and X are identical with respect to three phenyl rings, central bridge linking middle and third phenyl ring, left and right terminal end groups for the same homologue from series-1 to series-X. But they differ with respect to central bridges $-\text{CH}=\text{CH}-\text{COO}-$ and $-\text{COO}-$ linking first and middle phenyl rings for the same homologue from series-1 to series-X and Y. Thus, combine effect of molecular rigidity plus flexibility vary for the same homologue from series to series and from homologue to homologue in the same series. Homologous series 1 and Y are identical in all above respect except right handed tail end groups, $-\text{OC}_{16}\text{H}_{33}$ and $-\text{OC}_{14}\text{H}_{29}$ for the same homologue from series 1 to series Y and from homologue to homologue in the same series as a consequence of combined effect of molecular rigidity plus flexibility whose magnitudes decides the possibility of inducing mesomorphism and its thermotropic behaviours. Following table-3 represents some thermometric behaviours of presently investigated novel chalconyl homologous series-1 and the series-X [32] and Y [33] chosen for comparative study.

Table 3: Relative Thermal Stability in $^{\circ}\text{C}$

Series $\longrightarrow$	1	X	Y
smectic-isotropic or smectic-nematic Commencement of Smectic phase	—	—	—
Nematic-Isotropic Commencement of Nematic phase	99.75 (C_6 - C_{18}) C_6	123 (C_7 - C_{18}) C_7	113.14 (C_6 - C_{18}) C_6
Total upper and lower mesophase length range in $^{\circ}\text{C}$ C_i to C_j	19 to 34 C_6 to C_8	13 to 34 C_7 to C_{12}	10 to 26 C_8 to C_{10}

From above table-3 it is clear that

- Homologous series 1, X and Y under comparative study are nematogenic with absence of smectogenic property.
- The mesogenic property commences from C₆ (series 1 and Y) or C₇ (series X) homologue.
- Thermal stability depresses as unchanging tail n-alkyl chain is shortened.
- Thermal stability is enhanced if -CH=CH-COO- central bridge is replaced by -COO-.
- Thermal resistivity is poor for all the series under comparison.

The intermolecular suitable magnitudes of anisotropic end to end forces as a consequence of molecular rigidity and flexibility, due to the appropriate permanent dipole moment, dipole-dipole interactions etc. are sufficient and fit to facilitate and to induce nematogenic character only, but are insufficient to maintain focal conic networking molecular arrangement in rigid crystal and subsequent sliding layered molecular arrangement in floating condition under exposed thermal vibrations. Thus, smectogenic character fails to facilitate in all the series-1, X and Y under comparative study. The extent of molecular noncoplanarity for all the series 1, X and Y are equivalent but it slightly differs for the difference of -CH=CH- unit in case of series-X which, causes a little bit late (C₇) commencement from C₆ homologue for series 1 and Y. The observed difference for thermal stabilities is attributed to the unusual and unexpected molecular status of n-alkyl chain of both ended n-alkoxy terminals -OR and -OC₁₄H₂₉ depending upon resultant differing polarity difference of -OR and -OC₁₄H₂₉ which induces thermal resistivity, degree of mesomorphism and transition temperatures of homologues. Thus, thermal stabilities and upper and lower mesophase lengths ranges of series 1, X and Y are not much differed in magnitudes.

4. CONCLUSIONS

- Presently investigated chalconyl ester series is partly nematogenic whose degree of mesomorphism is shorter and of low melting type without exhibition of smectogenic property.
- The group efficiency order derived on the basis of (i) thermal stability (ii) early commencement of mesophase and (iii) higher degree mesomorphism are as under.

(i) **Smectic**

Series-1 = Series-X = Series-Y

Nematic

Series-X > Series-Y > Series-1

(ii) **Nematic**

Series-1 = Series-Y > Series-X

(iii) **Nematic**

Series-X > Series-1 > Series-Y

- Homologous series with longer n-alkyl chains bonded to third phenyl ring through oxygen atom as a tail end group are nematogenic only of low degree of mesomorphism.
- Difference of group polarities at the two terminal end groups decides the mesogenic behaviours of individual homologue and the same homologous series as a whole.
- Molecular rigidity and flexibility operates a phenomena of mesomorphism.
- Mesomorphism is very sensitive and susceptible to molecular structure.
- Mesogenic homologues of present study are useful for the study of binary systems for the manufacture of LC devices to be operated between 45⁰C to 50⁰C
- Chalconyl derivatives are bioactive molecules, which can be further studied for their bioactivity for the pharmaceutical and medicinal formulation.
- Present study supports and raises the credibility to the conclusions drawn earlier.

ACKNOWLEDGEMENT

Authors acknowledge thanks to Dr. R. R. Shah, principal of K. K. Shah Jarodwala Maninagar Science College, Ahmedabad. Authors thanks to Dr. A. V. Doshi, Ex-principal of M.V.M. Science and Home sc. College - Rajkot for his constant support, inspirations and help, as and when needed during the course of present investigation. Authors thank to NFDD Centre for the analytical and spectral services.

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